# NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

# TECHNICAL NOTE 3144

COEFFICIENT OF FRICTION AND DAMAGE TO CONTACT AREA

DURING THE EARLY STAGES OF FRETTING

II - STEEL, IRON, IRON OXIDE, AND GLASS COMBINATIONS

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### COEFFICIENT OF FRICTION AND DAMAGE TO CONTACT AREA DURING

THE EARLY STAGES OF FRETTING

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### SUMMARY

Experiments were conducted to study the start of fretting and the cause of damage during the early stages (up to 400 cycles) of fretting of steel-steel combinations at a frequency of 5 cycles per minute, an amplitude of 0.006 inch, a load of 150 grams, in air with relative humidity of less than 10 percent. Pure iron, glass, and iron oxide powder compacts were used in supplementary experiments. The results of microscopic observation of the contact area and measurements of coefficient of friction lead to these conclusions:

- 1. Fretting starts with severe adhesion between surfaces. The adhesion varies with the material combination, as shown by the initial coefficient of friction, but is of primary importance because it precedes and initiates the other phenomena observed.
- 2. In the early stages of fretting, several other wear phenomena in addition to adhesion occur. Their relative importance varies with the materials fretted, and they can occur simultaneously at different points within a contact area. They are:
  - (a) Plowing by protruding transferred material: The plowing is more pronounced when adhesion is greater.
  - (b) Formation of debris (loose fragments): Oxide debris is not evident when large wear fragments are produced, whereas oxide debris (Fe<sub>2</sub>O<sub>2</sub>) is evident in a very few cycles if the wear fragments are small.
  - (c) Formation of films by compacting small particles into clearances in the contact area: The formation occurs readily if one of the surfaces is hard. These films can plow the opposing metal surface.

#### INTRODUCTION

One of the more serious problems of wear existing in machines and metal structures is that of fretting. Fretting is the surface damage that occurs when contacting solids experience slight reciprocating slip. The wear, pitting, and debris caused by fretting, particularly in aircraft, can lead to loss of tolerance, increased fatigue susceptibility, and seizure. The later stages of fretting have been studied widely, but a lack of experimental evidence on the nature of the start and early stages of fretting remains. This lack of information has been a deterrent to the understanding of fretting, and has probably delayed finding a means of prevention or mitigation.

An investigation has been made of the start and early stages of fretting of copper (ref. 1). Only a few incidental observations (refs. 2 to 5) have been made on the start of fretting of steel in spite of its great practical importance. In these investigations of steel the start of fretting was associated with such phenomena as increase in cohesion, smearing, adhesion, and interlocking.

The research reported herein was conducted at the NACA Lewis laboratory to provide more experimental information about the start of fretting and the cause of damage during the early stages of fretting of steel against steel. Experiments were also conducted using pure iron, glass, and compacts of iron oxide to supplement the data obtained in the steel against steel experiments. Fretting was produced by reciprocating flat specimens in contact with convex specimens at a constant load, frequency, amplitude, and humidity. Clean, unlubricated specimens were used. A continuous record of friction force was made, and in specimen combinations using glass flats, the fretting was observed as it occurred. Debris was analyzed by chemical spot tests and surfaces were examined by electron diffraction.

# APPARATUS

The apparatus (fig. 1), designed to produce fretting at low frequency, is described in detail in reference 1. A flat specimen slides back and forth in contact with a convex specimen under a load of approximately 150 grams with an amplitude of 0.006 inch and a frequency of 5 cycles per minute. The relative humidity of the air surrounding the specimens during fretting was held to less than 10 percent. The humidity was measured by a hygrometer calibrated against a dew point potentiometer. Friction force was measured by means of a strain gage attached to a dynamometer ring and recorded by a photoelectric potentiometer. Normal load was measured for each run by determining the upward force required to separate the specimens. The accuracy of measurement of coefficient of friction (ratio of friction force to measured load) was estimated to be ±5 percent.

# MATERIALS AND PROCEDURE

Materials. - Three different steels were chosen as representing common engineering materials:

- (1) The convex specimens for all steel fretting runs were 1/2-inch-diameter commercial steel balls containing approximately 1 percent carbon and having a case hardness of Rockwell C-62.
- (2) Some flat specimens were made of a drill-rod stock containing approximately 1 percent carbon and were either hardened to Rockwell C-60 or annealed to less than Rockwell B-80.
- (3) Other flat specimens were made of an unhardened tool steel containing approximately 1 percent carbon and 1.45 to 1.55 percent manganese and having a hardness of Rockwell B-90.

The pure iron specimens were made from Puron stock (99.97 percent Fe) hardened to Rockwell B-82 by cold working; this material was chosen for simplicity, homogeneity, and established friction characteristics.

The glass specimens were microscope slides. Chemically pure powders of ferric oxide  $\text{Fe}_2\text{O}_3$  and ferrosoferric oxide  $\text{Fe}_3\text{O}_4$  were obtained commercially.

Specimen preparation. - All specimens except glass were abraded on 2/0 emery paper to give a uniform surface finish of 10-20 microinches root mean square. Consistent and thorough cleaning of specimens to remove the last trace of grease was important for reproducibility. Freedom from grease was indicated by high initial values of coefficient of friction  $\mu$ . Details of the cleaning procedures for metal and glass are given in appendix A.

The oxide specimens were machined from compacts made by pressing and sintering pure  $\rm Fe_2O_3$  and  $\rm Fe_3O_4$  powders under controlled conditions (appendix B).

Experimental procedure. - The cleaned specimens were mounted in the specimen holders of the apparatus and the load applied by adding weights. The cover of the Lucite box was put in place and dry air was started flowing through the enclosure. When the relative humidity of the escaping air had dropped to 10 percent, the reciprocating action was started. In the case of glass flat specimens, microscopic observations (X75 to X200) of the fretting action as it occured were correlated with the friction tracing. For metal-metal combinations, the surfaces were separated and examined microscopically after runs lasting 1/2, 1, 5, 10, 20, 50, 100, 200, 300 or 400 cycles. New specimens were used for each run.

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Small quantities of debris were identified by chemical spot tests (appendix C). Electron diffraction was used to determine the chemical composition of surfaces before the runs, and also to identify debris when possible.

#### RESULTS

Coefficient of friction was plotted against cycles for each run and all the data obtained are presented in figures 2, 4, 5, and 7. The friction curve presented is a smoothed curve drawn through the average of all the runs. Correlation of number of cycles, observation of contact area, and coefficient of friction for various specimen combinations is presented in tables I to IV.

The contact area over which wear was observed was quite small, varying from 0.002 to 0.02 inch in length and width. The phenomena observed during fretting occur in the sequence shown in the tables, but the phenomena can be in different stages at various parts of the contact area at the same time. All chemical compositions given parenthetically in either the tables or the text were identified by chemical spot tests conducted as described in appendix C.

# Fretting of Steel

Steel against glass. - Hard 1-percent-carbon steel convex specimens were fretted against glass flats so that the phenomena which cause the fretting of steel could be observed as they occurred. Examination of table I and figure 2 shows that the first sliding motion produces material transfer and a high coefficient of friction (> 1.2). The formation of and increase in amount of film (Fe<sub>2</sub>0<sub>3</sub>) on the glass and a small amount of plowing of the steel by thickened portions of the film effected a reduction in the value of  $\mu$  to a minimum of 0.60 at 20 cycles. The detachment of the film from the glass and the appearance and accumulation of rust-colored debris (Fe<sub>2</sub>0<sub>3</sub>) in and around the contact area are associated with a rise in the value of  $\mu$  by 200 cycles to a stable state in which the value of  $\mu$  is approximately 0.75.

Steel against steel. - The results of fretting hard 1-percent-carbon steel convex specimens against hard and soft (annealed) 1-percent-carbon steel flats and soft tool steel flats may be seen in table II and figure 3. (The individual data points in figure 3 have been omitted for clarity.) In all steel against steel runs, fretting started with a value of  $\mu$  of 0.60 to 0.70. The presence of a film after the first few cycles of fretting was also observed. The increase in the

quantity of film and a small amount of plowing were associated with a reduction in friction from the higher values to 0.53 to 0.61 in 5 to 20 cycles. The film formation and plowing were followed by the appearance of loose particles, some of which were trapped in the contact area. The value of  $\,\mu\,$  increased slowly to 0.58 to 0.65 by 300 cycles.

Softer specimens always suffered more pronounced plowing. In addition, loose rust-colored debris ( $Fe_2O_3$ ) appeared within 10 cycles in the case of hard steel-hard steel combinations, but not until after 100 cycles in the case of hard steel - soft tool steel combinations. However, the rate of wear as estimated from the change in the area of the wear spot was found to be the same for all three steel specimen combinations.

Comparison of the curves in figure 3 shows that over the first 300 cycles the stable values of  $\mu,$  even though within experimental error, are slightly lower with greater hardness of the flat specimen. When the flat is annealed (< Rockwell B-80), the stable  $\mu$  is about 0.65; for the tool steel flat (Rockwell B-90), the stable  $\mu$  is 0.61 to 0.64; and for the hardened (Rockwell C-60) steel flats,  $\mu$  is 0.56 to 0.58.

# Fretting of Pure Iron

Pure iron against glass. - Pure iron convex specimens were fretted against glass flats to allow observation of the phenomena as they occurred and to permit comparison with hard steel-glass fretting. shown in table III and figure 4, the first half cycle of motion caused transfer of iron (Fe) to the glass, and a coefficient of friction of >0.75. The transferred metal plowed furrows in the surface from which it came, and the coefficient of friction was reduced to a minimum value of 0.66 by 10 cycles. The plowing action was followed by a loosening of the fragments adhering to the glass and the accumulation of black metallic debris and film formation on the metal. (The adhesion of iron fragments to the glass in the fretting of pure iron against glass was not so great as the adhesion of the oxide film transferred in the fretting of steel against glass. Detachment of the iron fragments occurred within 10 to 20 cycles, whereas the oxide film did not break up for 50 to 60 cycles.) Coefficient of friction increased to a stable value of 0.83 by 240 cycles, and small amounts of rust-colored debris (Fe $_2$ 0 $_3$ ) appeared in the black debris after 300 cycles.

Pure iron against pure iron. - Pure iron was fretted against pure iron in order to observe the wear resulting on a homogeneous material basic to steel, and the results are presented in table IV and figure 5.

The first phenomenon to occur was mutual plowing, and it produced a coefficient of friction which varied from 0.52 to 1.02, depending on the ultimate degree of cleanliness of the surfaces, but which averaged 0.82. The value of  $\mu$  in each run then increased to an average peak value of 0.94. Subsequently, the reciprocating action produced loose iron fragments, at first large and then small, and a decrease in the value of  $\mu$  to a minimum of 0.6 at 25 cycles. Some of the small fragments appear to be trapped in the contact area and compacted into films, whereas other fragments accumulate outside the contact area as loose black metallic debris. After 100 cycles, brown film appears in the contact area, and the value of  $\mu$  increases slightly to a stable 0.64. After 300 cycles, traces of rust-colored debris (Fe<sub>2</sub>O<sub>3</sub>) appear in the black metallic debris.

A photomicrograph of a 15:1 taper section of the damaged area on pure iron after fretting against pure iron for 400 cycles is shown in figure 6. The presence of black debris on the surface and severe deformation of the metal below the surface are evident. The debris is not imbedded in the deformed metal as was the case for copper (ref. 1, fig. 4).

# Fretting of Iron Oxide Powder Compacts Against

# Iron Oxide Powder Compacts

 $Fe_2O_3$  compacts against  $Fe_2O_3$  compacts. - Compacts of  $Fe_2O_3$  were fretted against  $Fe_2O_3$  compacts to observe fretting and frictional behavior in the absence of metal-to-metal contact. Figure 7 shows that the value of  $\mu$  started at 0.60 and reduced to 0.51. The action produced an abraded flat spot, portions of which showed burnished films of oxide and around which was rust-colored debris that appeared to be the same as that found in the fretting of steel.

 $\rm Fe_3O_4$  compacts against  $\rm Fe_3O_4$  compacts. - In order to study the oxidation occurring during fretting as well as fretting and frictional behavior in the absence of metal-to-metal contact,  $\rm Fe_3O_4$  compacts were fretted against  $\rm Fe_3O_4$  compacts. Figure 7 shows that the value of  $\mu$  started at 0.30, peaked at 0.36 by 5 cycles, decreased to a minimum of 0.29, and then slowly increased to 0.45 at 320 cycles and to 0.50 at 600 cycles. The action produced a flat spot which showed oxide films plus the same rust-colored debris observed from steel and from  $\rm Fe_2O_3$  compact fretting.

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In short, the Fe<sub>3</sub>O<sub>4</sub> compacts fretted to produce Fe<sub>2</sub>O<sub>3</sub> debris. In a few runs (not presented), the Fe<sub>3</sub>O<sub>4</sub> compacts did not fret but maintained a relatively low constant  $\mu$  value near O.15. No debris was found; in fact the wear area was hardly discernible microscopically.

The initial friction value for  ${\rm Fe_3O_4}$  compacts which fretted and the constant friction value for  ${\rm Fe_3O_4}$  compacts which did not fret suggest that the coefficient of friction for  ${\rm Fe_3O_4}$  is between 0.15 and 0.3 at these slow sliding speeds.

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The results obtained with both oxide compacts show formation of the final iron oxide, Fe $_2$ O $_3$ , during fretting. The results further show the stable value of  $\mu$  obtained during the fretting of oxide compacts (0.5 to 0.53) to be lower than those obtained for steel against steel (0.58 to 0.65). This difference is possibly a result of the persistence of metal contact during the fretting of steel in stages when oxide has accumulated on the surface.

# DISCUSSION

# Phenomena Observed During Fretting

A comparison of results obtained with the various specimen combinations of this investigation and with copper in reference 1 indicates that low amplitude reciprocating sliding produces not just one but several phenomena: adhesion, plowing, formation of debris, and formation of films. These phenomena, all of which contribute to fretting damage in its early stages, will be discussed separately.

Adhesion. - The occurrence and extent of adhesion or cold welding was indicated by transfer of material during the first few cycles of fretting of practically all material combinations. Copper suffered the greatest adhesion and transferred in large amounts to glass, copper, and steel, whereas iron transferred in slightly smaller amounts to glass and to pure iron. The start of fretting of steel showed considerably less adhesion. In the case of hard steel against glass, only a film of oxide was found on the glass. Probably small metal fragments transferred initially but quickly oxidized. For steel against steel a film was observed but could not be identified.

The extent of adhesion was also indicated by the initial value of  $\mu$  obtained, the values ranging from 0.6 for hard steel against hard steel to greater than 1.2 for copper against glass.

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Adhesion is considered to be the most important phenomenon observed in these unlubricated fretting runs because it precedes and initiates the other phenomena. Its elimination or minimization should prevent or reduce fretting damage.

Plowing. - The second phenomenon observed was plowing of a surface by material adhering to the opposing surface. Damage by plowing was greatest in the case of copper, a material inherently susceptible to galling. Protruding welded fragments plowed large deep furrows at the start of fretting. In the case of iron, plowing occurred first by welded fragments and later by thick films, but was less than for copper. In the case of steel, damage due to plowing was less evident (probably because the furrows were smaller, like scratches, and were more numerous), and plowing occurred only after films (adherent oxide) had formed. Plowing of a steel surface by film was greater if the opposing surface was glass or hard steel.

The reduction in values of  $\mu$  during fretting was usually greatest (for example, 1.65 to 0.6 for copper against glass) when material transfer and plowing were initially pronounced.

Formation of debris. - The nature and quantity of debris (loose fragments) varied with the materials studied. Copper and iron produced a few large metallic fragments initially, smaller metallic fragments later, and then a small quantity of oxide powder. Steel produced a larger quantity of debris composed of oxide powder, with possibly a few small metallic particles. No large particles, either metallic or oxide, were found with steel. These results suggest that only small wear particles can oxidize completely, and that fretting of steel produced very small metal particles which oxidized completely.

Although iron has a greater affinity for oxygen than copper, fretting produced oxide powder on copper within 100 cycles, but on iron not until after 300 cycles and only in traces.

In most cases where identification was possible, the oxide formed was found to be the final oxidation state of the material, that is,  $Fe_2O_3$  for iron, steel, and iron oxide compacts, and CuO for copper and CuO compacts. The only exceptions were  $Cu_2O$  compacts, which fretted to give  $Cu_2O$  powder debris.

Formation of films. - The formation of films by compacting of small fragments into local clearances in the apparent contact area was observed, and under the conditions of these experiments preceded the appearance of loose oxide powder. The films were shades of brown for iron and steel and were much thicker than films formed by heating metals in air. They formed on iron after 50 cycles of fretting and on hard

steel in the first half-cycle, particularly when the steel was fretted against glass or hard steel. Compacts of iron oxide powder also formed films during fretting. No film was visible when copper fretted against copper, but a greenish film formed on glass when it fretted against copper. Thus films form readily during fretting, and the formation is enhanced when the opposing surface is a hard material. Films probably form in other types of wear in the same manner, but in fretting the formation is aided by the low-amplitude reciprocating motion.

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The ability of oxide particles to form a film is unusual in view of their commonly accepted hardness or lack of plasticity. However, supplementary experiments have shown that fine dry Fe<sub>2</sub>O<sub>3</sub> powder collected from fretting of steel against glass can be pressed to form a film, and further verification was found in reference 6 where Stott observed the flattening of oxide nodules during experiments with jewel bearings.

Proposed Mechanism of Fretting of Steel Against Steel

The following description of the fretting of steel against steel is proposed from the results that have been presented herein and from observations by other investigators (refs. 7 to 9).

Contact of loaded surfaces causes elastic and plastic deformation of contacting asperities (ref. 7, p. 19). This deformation causes interlocking (ref. 8) and rupture (ref. 7, ch. I), or both, of the thin (<100 A, ref. 7, p. 149) oxide film normally found on a steel surface. The rupture of the film permits metal-to-metal contact and thus adhesion, or cold-welded junctions, and relatively high coefficients of friction. With sliding, welded or interlocked junctions are broken at some weak section, metal transfer may occur (ref. 7, plate IX of fig. 1), and some loose metal and oxide fragments are formed. The fragments quickly oxidize because of their small size, increased temperature (ref. 7, ch. II), and high state of stress (ref. 9). A discontinuous film is formed by compacting of the oxide fragments and the film increases in coverage and thickness with continued reciprocating sliding. This increasing coverage reduces the coefficient of friction.

As the films thicken, they begin to plow furrows in the opposite metal surface. This was deduced from the shape and position of a spot of film in comparison with a furrow in the opposite surface, and was observed directly when glass flats were used. The deepening of the furrow shifts the load to another part of the contact area, and another furrow is formed.

The relief from load that the first contact experiences as a result of the presence of the furrow permits the film to disintegrate or

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slough off and it appears as rust-colored debris just beyond the end of the furrows. The reciprocation of the sliding during fretting causes debris to collect on the floors and sides of the furrows. The amount that can collect, however, reaches a maximum and clean areas remain which permit metal-to-oxide contact and possibly metal-to-metal contact. In later stages of fretting, not investigated in the research reported herein, a "crust" of oxide could form and wear would probably then be of the abrasive type described in reference 5.

# SUMMARY OF RESULTS

The results of microscopic observation of the low-frequency fretting of iron and steel and the measurements of coefficient of friction are summarized as follows:

The fretting of steel against steel started with coefficients of friction between 0.60 and 0.70. This was followed by formation of films, some plowing, production of loose  $\mathrm{Fe_2O_3}$  debris, and a reduction in the coefficient of friction to between 0.53 and 0.61. An essentially stable coefficient of friction was reached after approximately 20 cycles, giving values which rose slowly to between 0.58 and 0.65 at 300 cycles.

The supplementary experiments conducted with glass, iron, and iron oxide produced the following results:

- 1. Glass flats showed that films were formed by compacting of fragments and that these films could plow an opposing metal surface. Coefficient of friction values obtained when glass was one of the specimens were generally higher than those obtained with metal-against-metal combinations.
- 2. Fretting of pure iron against pure iron produced mass welding and subsequently metallic fragments and severe plowing. The coefficient of friction was initially 0.82, reduced to 0.60 in 25 cycles, and after 100 cycles reached an essentially stable value of 0.64 as metallic debris accumulated.
- 3. Iron oxide compacts, both  $\mathrm{Fe_2O_3}$  and  $\mathrm{Fe_3O_4}$ , fretted to form  $\mathrm{Fe_2O_3}$  debris the same as that found in fretting of steel. The coefficient of friction for  $\mathrm{Fe_2O_3}$  compacts against  $\mathrm{Fe_2O_3}$  compacts was initially 0.60 and within 100 cycles reduced to an essentially stable value near 0.51. The coefficient of friction of  $\mathrm{Fe_3O_4}$  compacts fretting against  $\mathrm{Fe_3O_4}$  compacts began at 0.30, then slowly increased to about 0.50 after 600 cycles.

# CONCLUSIONS

- 1. Fretting starts with severe adhesion. The adhesion varies with the material combination, but is of primary importance because it precedes and initiates the other phenomena observed.
- 2. In the early stages of fretting, several other wear phenomena occur. Their relative importance varies with the materials fretted, and they can occur simultaneously at different points within a contact area. They are:
  - (a) Plowing by protruding transferred material. The plowing is more pronounced when adhesion is greater.
  - (b) Formation of debris (loose fragments). Oxide debris is not evident when large wear fragments are produced, whereas oxide debris is evident in a very few cycles if the wear fragments are small.
  - (c) Formation of films by compacting small particles into clearances in the contact area. The formation occurs readily if one of the surfaces is hard.

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### APPENDIX A

#### CLEANING OF SPECIMENS

General precautions. - Certain precautions were necessary to obtain and maintain grease-free specimens. All solvents used (water, benzene, and 100 percent ethyl alcohol) were redistilled. All vessels used were of pyrex and were cleaned in sulfuric acid-sodium dichromate cleaning solution, rinsed with tap water and redistilled water, and dried in an oven. All specimen and vessel handling tools were cleaned by heating to redness.

Cleaning or metal specimens. - The iron and steel specimens were cleaned anodically by the following procedure:

- (1) Washed in uncontaminated naptha
- (2) Rinsed at least 10 times with benzene in a Soxhlet extractor
- (3) Rinsed at least 10 times with ethyl alcohol in a Soxhlet extractor
- (4) Dried with air blower
- (5) Cleaned anodically in a solution composed of 2 percent NaOH and 10 percent Na<sub>2</sub>CO<sub>3</sub> at a temperature between 80° and 90° C with a current density of about 0.3 ampere per square inch. Cleaning time was about 1 minute.
- (6) Quickly rinsed in water
- (7) Rinsed with alcohol and dried with air blower

This procedure gave specimens that were grease-free as shown by high initial coefficients of friction and bore only a trace of oxide as shown by electron diffraction examination.

Cleaning of glass specimens. - The glass microscope slides were scrubbed under nitric acid with glass wool, rinsed with tap and redistilled water, and dried in oven.

# APPENDIX B

# COMPACTING OF IRON OXIDE POWDERS

Compacts of  ${\rm Fe}_2{\rm O}_3$  and of  ${\rm Fe}_3{\rm O}_4$  were made by pressing and sintering the respective powders. The  ${\rm Fe}_2{\rm O}_3$  powder was pressed in a hydrostatic die at 30,000 pounds per square inch and sintered in a helium atmosphere at 1500° F for 7 hours. The  ${\rm Fe}_3{\rm O}_4$  was pressed in a hydrostatic die at 30,000 pounds per square inch and sintered in vacuum at 2400° F for 2 hours. X-ray diffraction patterns taken of these compacts showed that the chemical composition was unchanged. The  ${\rm Fe}_2{\rm O}_3$  compact was purple colored, relatively coarse-textured, and soft when compared with the black  ${\rm Fe}_3{\rm O}_4$  compact which was dense and very hard.

## APPENDIX C

#### CHEMICAL SPOT TESTS

The chemical spot tests were conducted under a blanket of  ${\rm CO}_2$ , were illuminated with a cool light, and were sometimes observed with a low-power stereomicroscope. The identifications were based on the following facts: (1) In a solution of slightly acidic copper sulfate, copper will be plated on metallic iron but not on iron oxides, (2) iron Fe and ferrous oxide FeO dissolved in concentrated HCl give only ferrous ions Fe<sup>++</sup>, (3) ferric oxide (Fe<sub>2</sub>O<sub>3</sub>) dissolved in HCl gives only ferric ions Fe<sup>++</sup>, (4) whereas ferrosoferric oxide Fe<sub>3</sub>O<sub>4</sub> or FeO·Fe<sub>2</sub>O<sub>3</sub> when dissolved in acid gives both ferrous and ferric ions.

The test procedure required three samples of the unknown and was as follows:

# Sample A.

1. Add few drops of approximately 0.1 N copper sulphate solution to the unknown. The appearance of copper indicates presence of iron.

# Sample B.

l. Dissolve the unknown with a drop of concentrated hydrochloric acid, and add drop of  $\alpha,\alpha$ -dipyridal solution. Appearance of red color indicates presence of ferrous ions. This color is more pronounced if acidity is reduced with sodium acetate.

# Sample C.

1. Dissolve the unknown with a drop of concentrated hydrochloric acid, dilute with two or three drops of water, and add one drop of potassium thiocyanate. The appearance of blood red color indicates presence of ferric ions.

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TABLE I. - FRETTING OF HARD, 1-PERCENT-CARBON STEEL AGAINST GLASS

Number of cycles	Phenomena observed	Coefficient of friction, µ
1/2	First motion produces several parallel rows of small brown spots transferred to glass and also cracking of glass.	>1.2
1 to 10	Size and number of spots and number of rows increase to form a continuous brown film on glass (Fe <sub>2</sub> O <sub>3</sub> ).	>1.0 to 0.65
10 to 20	Thickened portions of film, formed by compacting of fragments against glass, plow steel to form shallow furrows.	0.65 to 0.6
20 to 60	Rust-colored loose debris appears outside of contact area. Parts of film cease to move with glass, apparently adhering to metal surface. Small loose particles are visible in and around shallow furrows.	0.6
60 to 200	Steel contact area is plowed by a few spots of thick film still adhering to glass. They disappear as areas of actual contact move from one location to another.	0.6 to 0.75
200 to 400	Amount of film in contact area becomes essentially constant whereas amount of rust-colored debris outside of contact area steadily increases. Film on glass plows poorly defined furrows. Particles trapped in contact area are "worked" by reciprocating action.	0.75 to 0.77

TABLE II. - FRETTING OF STEEL AGAINST STEEL

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Number of cycles	fretting	Coefficient of friction, µ Hard 1-percent-carbon steel convex against:		
		Hard 1- percent-carbon steel flat	Soft 1- percent-carbon steel flat	Soft tool steel flat
1/2	Small amount of brown film is on convex specimen fretted against tool steel, but no change on convex fretted against hard or soft 1-percent-carbon steel.  Flats are lightly scratched.	>0.6	>0.7	<b>&gt;</b> 0.63
1	Thin patches of film(unidentified) are on contact area of both specimens.  Flats have less film and are scratched.	0.59	0.69	0.63
5	More film is on convex, and shallow furrows are on flat contact area.	0.58	0.65	0.6
10	Loose rust-colored debris is found around contact area of hard-hard steel combination. Displaced material is found at one end of furrow on soft 1-percent-carbon steel flat. All combinations show more film.	0.56	0.63	0.6
20 to 30	Position of film on convex matches shallow, poorly defined furrow on hard steel flat. Loose dark fragments are found on contact areas of soft steel flats.	0.53	0.61	0.6
<b>4</b> 0 <b>to</b> 60	Loose rust-colored debris (Fe <sub>2</sub> 0 <sub>3</sub> ) appears around contact area on soft 1-percent-carbon steel flat. The 1-percent-carbon steel com- binations also have black debris on and around furrows, and por- tions of debris are piled up and compacted in contact area.	0.5 <u>4</u>	0.62 to 0.63	0.6
100	Loose rust-colored debris is found on tool-steel flat.	0.55	0.64	0.6
200 and 300	More poorly defined furrows and accumulation of rust-colored and black debris around contact area of all specimens. Chemical tests reveal only Fe <sub>2</sub> O <sub>3</sub> in debris. Soft steel flats are plowed more and have a greater amount of loose debris in contact area. Very thick films or crust cover contact area of 1-percent-carbon steel - tool steel combination.	0.56 to 0.58	0.66 to 0.65	0.61 to 0.63

TABLE III. - FRETTING OF PURE IRON AGAINST GLASS

Number of cycles	Phenomena observed	Coefficient of friction, µ
1/2	Large fragments (Fe) adhere to and are trans- ferred to glass.	<b>&gt;</b> 0.75
1 to 10	Fragments moving with glass plow furrows in metal surface from which they came. Number of fragments and furrows increases with each half cycle.	0.75 to 0.66
10 to 20	Fragments, which do not adhere very strongly, are scraped off glass and transferred back and forth between metal and glass.	0.66 to 0.67
20 to 50	Spots of brown film (Fe <sub>2</sub> O <sub>3</sub> ) appear on glass. Film is very tenacious compared with transferred iron fragments. Loose black debris, which appears to be metallic, slowly appears as fragments are "worked" out of contact area.	0.67 to 0.72
50 to 300	Film appears on metal contact area. Spots remaining on glass change both in position and in degree of activity. Material forming spots plows furrows in metal.	0.72 to 0.83
300 and greater	Amount of film adhering to metal contact area becomes essentially constant. Rust-colored powder (Fe <sub>2</sub> O <sub>3</sub> ) appears in some runs in black metallic debris outside contact area.	0.83

TABLE IV. - FRETTING OF PURE IRON AGAINST PURE IRON

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Number of cycles	Appearance of contact area after fretting	Coefficient of friction,
1/2	Furrows are plowed in both surfaces by large pro- truding metal fragments. Floors of furrows are smooth and clean.	0.82
5	Smaller loose black metallic fragments are visible in and around broadened furrows.	0.9 to 0.8
10	Damage is similar to but more extensive than that observed at 5 cycles. Floors of furrows are speckled with small fragments.	0.8 to 0.7
20	Accumulation of fragments is found across furrows. Contact area is generally flatter and broader.	0.65 to 0.60
50	Discontinuous colorless film, observed by vertical illumination, is found in contact area. Black debris has accumulated outside contact area.	0.63
100,	Brown film is found in contact area. Black debris is occasionally found compacted to form plateaus.	0.64
300, 400	Greater amounts of black debris and occasionally a trace of rust-colored debris (Fe <sub>2</sub> O <sub>3</sub> ) are found outside contact area. Wear area is covered by brown to black film which varies in thickness.	0.64

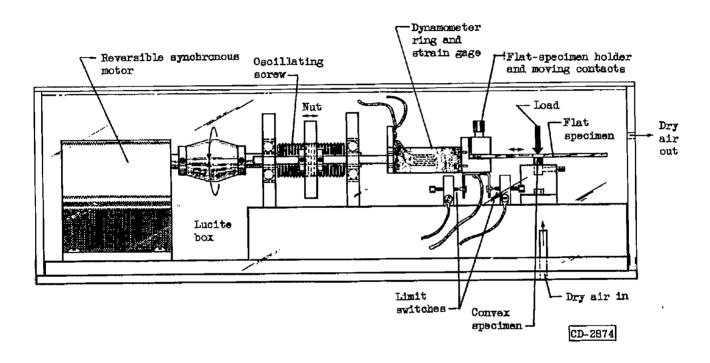


Figure 1. - Fretting apparatus.



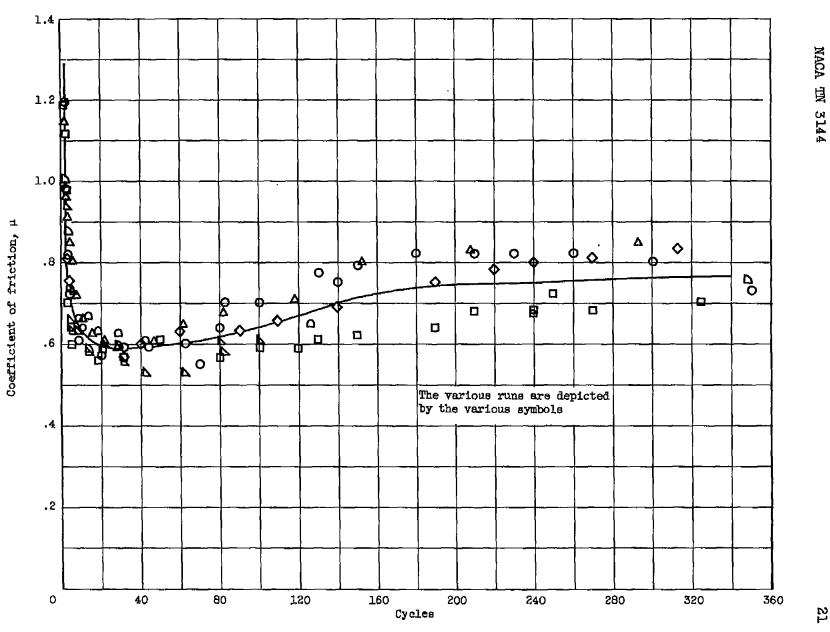


Figure 2. - Friction during fretting of hard 1-percent-carbon steel against glass.

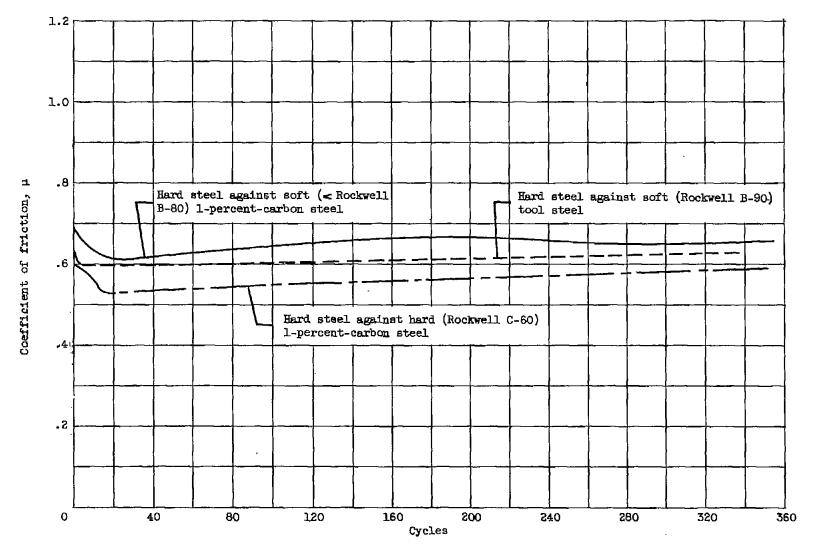


Figure 3. - Friction during fretting of hard (Rockwell C-62) 1-percent-carbon steel against other steels.

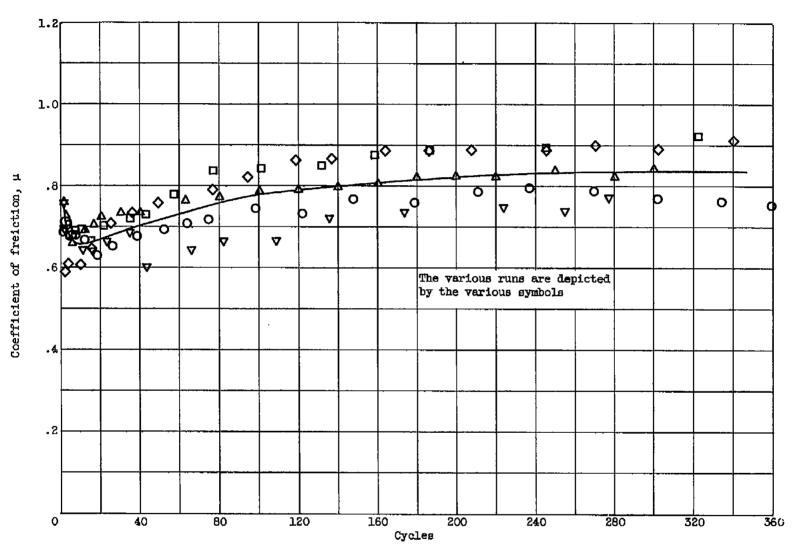


Figure 4. - Friction during fretting of pure iron against glass.

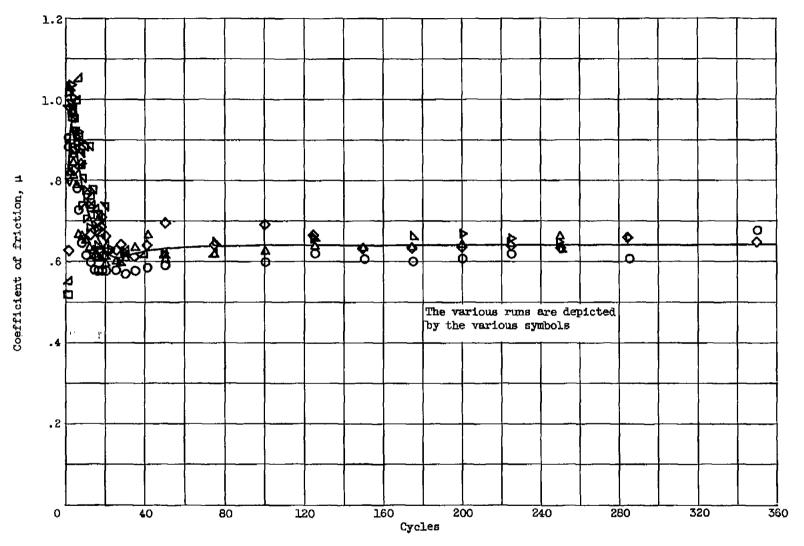


Figure 5. - Friction during fretting of pure iron against pure iron.

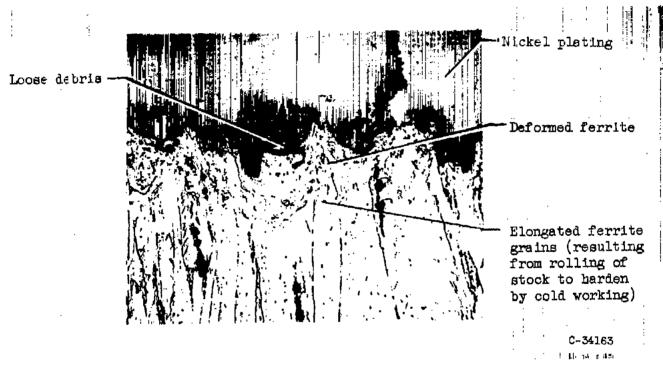


Figure 6. - Photomicrograph of taper section of fretted area of pure iron specimen fretted against pure iron for 400 cycles. Horizontal magnification, X500; vertical magnification, approximately X7500.

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Figure 7. - Fretting of iron oxide compacts.